D.5 AIR TESTING

D.5.1 Negative and Positive Controls

a) Negative Controls

1. Method Blanks – Shall be performed at a frequency of at least one (1) per batch of twenty (20) environmental samples or less. The results of the method blank analysis shall be used to evaluate the contribution of the laboratory provided sampling media and analytical sample preparation procedures to the amount of analyte found in each sample. If the method blank result is greater than the detection limit and contributes greater than 10% of the total amount of analyte found in the sample, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If the source of the contamination is found to be ambient background, the data will be qualified in the report.

2. Break Through – If sampling trains are composed of multiple sampling media sections, such as the "front "and "back" sections of sorbent tubes, these sections shall be analyzed separatelyto document the sampling media collection efficiency. The laboratory shall have an standard operating procedure(s) for defining and detecting breakthrough. Samples with analytes that exhibit breakthrough shall be flagged as minimum amounts.

b) Positive Controls

I. Laboratory Control Sample – Shall be analyzed at a rate of at least one (1) per batch of twenty (20) or fewer samples for each analyte. If a spiking solution is not available, a calibration solution whose concentration approximates that of the samples, shall be including in each batch. If the target analyte concentrations are above the calibration midpoint, the LCS should be above the calibration midpoint. If the target analyte concentrations are below the calibration midpoint, the LCS should be below the calibration midpoint and if the target analyte concentrations vary across the entire calibration range, the sequential LCS concentrations shall vary across the entire calibration range.

II. Desorption Efficiency (Recovery) – Desorption efficiencies shall be determined for each analyte on each lot of sampling media used to collect that analyte. The laboratory shall have SOPs defining these procedures.

III. Surrogates - Shall be used as required by the test method.

IV. Matrix spike – Shall be used as required by the test method.

D.5.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates – Shall be analyzed at a minimum of 1 in 20 samples per sample batch. Analysis duplicates shall be used if laboratory duplicates are not available. The laboratory shall document their procedure to select the use of appropriate types of duplicate. The selected samples(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

5. Demonstration of Capability – (Sections 5.6.2 and 5.10.2.1) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, matrix, or test method.

6. Calibration – Calibration protocols specified in Section 5.9.4 shall be followed.

7. Proficiency Test Samples – The results of such analyses (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.5.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., MDL. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

a) A detection limit study is not required for any component for which spiking solutions are not available such as temperature or on-line analyses.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) Detection limits must be determined each time there is a significant change in the test method or instrument type.

d) It is essential that all sample processing steps of the analytical method be included in the determination of the detection limit.

e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to tie detection limits with quantitation limits.

D.5.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.5.6 Quality of Standards and Reagents

8. The source of standards shall comply with 5.9.2.

9. The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.

c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

D.5.7 Selectivity

The laboratory shall develop and document acceptance criteria for test method selectivity such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

D.5.8 Constant and Consistent Test Conditions

1. The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

10. The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.

c) If supplied or used bye the laboratory, procedures for field equipment decontamination shall be developed and their use documented.

d) The laboratory shall have a documented program for the calibration and verification of sampling equipment such as pumps, meter boxes, critical orifices, flow measurement devices and continuous analyzers, if these equipment are used or supplied by the laboratory.